PCT WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 : G01N 33/04, 33/02, 21/09 // 21/35

A1

(11) International Publication Number: WO 98/20338

(43) International Publication Date: 14 May 1998 (14.05.98)

(21) International Application Number: PCT/DK97/00492

(22) International Filing Date: 31 October 1997 (31.10.97)

(30) Priority Data:
1221/96 1 November 1996 (01.11.96) DK

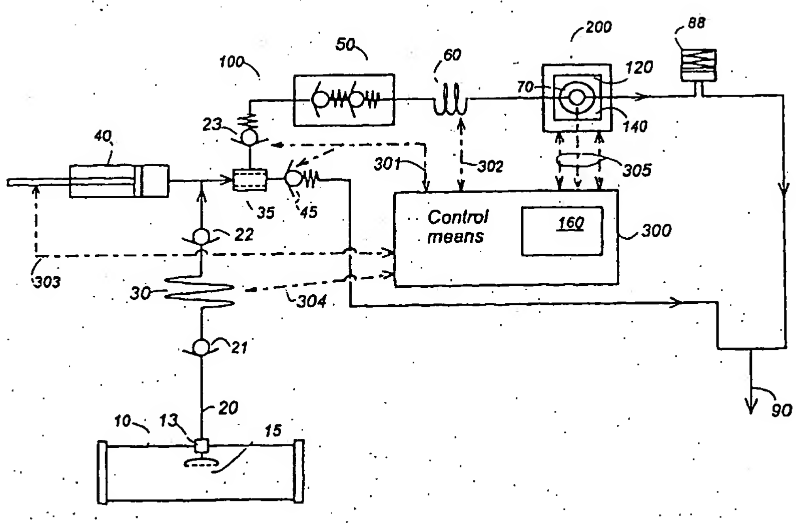
(71) Applicant (*for all designated States except US*): FOSS ELECTRIC A/S [DK/DK]; P.O. Box 260, DK-3400 Hillerød (DK).

(72) Inventors; and
(75) Inventors/Applicants (*for US only*): FRANDSEN, Andreas, Skærlund [DK/DK]; Parkvej 4, DK-3550 Slangerup (DK). PEDERSEN, Søren, Christian [DK/DK]; Brickasvej 21, DK-3400 Hillerød (DK). RIDDER, Carsten [DK/DK]; Højstrupvej 165, DK-2700 Brønshøj (DK). THOMSEN, Henrik [DK/DK]; Gl. Holmegårdsvej 710, DK-3400 Hillerød (DK).

(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ARIPO patent (GH, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).

Published
With international search report.

(54) Title: A METHOD AND FLOW SYSTEM FOR SPECTROMETRY AND A CUVETTE FOR THE FLOW SYSTEM



(57) Abstract

The present invention relates to an on-line method and a flow system as well as a cuvette for carrying out IR spectrometry for analysis of liquid food products, possibly containing dissolved gases, in a process line in a liquid food product processing plant, especially a dairy processing milk and milk products. A liquid sample is extracted from the process line to a measuring branch, the sample is thermostated and passed to a measurement cuvette. The IR-absorbance spectrum is measured, e.g. in the MID-IR or NIR-range. In order to obtain an on-line monitoring of the process line the liquid food sample is extracted directly from the process line into the measurement branch, in which the pressure is maintained at least as high as in the adjacent process line. The high pressure ensures that dissolved air will stay dissolved in the liquid food. Before each new sample the measurement branch and cuvette are flushed by high flow rates with a part of the new sample to clean the cuvette. The measurement cuvette has strong windows, preferably diamond windows to stand a high pressure and

TITLE OF INVENTION:

A METHOD AND FLOW SYSTEM FOR SPECTROMETRY AND A CUVETTE FOR THE FLOW SYSTEM

TECHNICAL FIELD

The present invention relates to a method and a flow system for carrying out spectrometry for analysis of a liquid food product, possibly containing dissolved gases, in a process line in a liquid food product processing plant, especially milk and milk products in a dairy, and comprising the following steps: 1) providing a liquid food sample from the process line to a measuring branch, 2) thermostating the liquid food sample, 3) passing the thermostated liquid food sample to a sample cuvette, 4) measuring at least part of the absorbance spectrum of the liquid food sample in the sample cuvette. The invention also relates to a measurement cuvette for the flow system. The present invention is specifically intended for IR-measurements, e.g. MID-IR and/or NIR-measurements for a determination of the quantities of specified components in the liquid food product.

BACKGROUND ART

A presently used method includes providing a milk sample from the process plant in an open sample container or cup from which dissolved gases may escape, passing part of the degassed milk sample from the container through a measurement branch into a measurement cuvette, performing the test and passing the tested milk sample to a waste outlet.

The presently used test instrument includes generally a flow system, an IR spectrophotometer, and a computer comprising a PC with harddisc, floppy disc drive, monitor and keyboard.

Instead of the above mentioned method it would be preferable to perform the test on-line and in-line in the process plant, and preferably in such a way that it also would be possible to let the tested milk sample be returned into the process line, to avoid the waste.

US 5,137,738 discloses a system and a method for controlling the butterfat content of milk. The entire product stream is monitored continuously by the use of optical density sensors. The preferred sensors have stainless steel bodies and housings med Pyrex windows and mount directly on the product output lines and operate at full flow and pressure. US 4,144,804 discloses a photoelectric monitoring system for continuously measuring the butterfat content of a sample of homogenized milk as the latter flows continuously through the processing system under the pressure of the homogenizer in the system. The known on-line sensors for milk products do not apply spectrometric analysis of the content.

flushing the cuvette with part of the recently (latest) extracted liquid food sample, further having provided that the cuvette having windows of a pressure and wear resistant material, being especially resistant to mechanical and chemical influences of the kind appearing in dairies. The flushing is performed under a pressure of from 100 - 200 bars, preferably from 110 - 150 bars across the cuvette, so the flushing rate will be high enough to ensure a thorough removal of the old sample including a cleaning of the cuvette.

The high pressure ensures that dissolved air will stay dissolved in the liquid food. The pressure and wear resistant materials, preferably diamond windows, allow the cuvette to stand high pressures and high flow rates. Other window materials might break or move causing the cuvette to widen and thereby causing the IR transmission loss through the cuvette to raise, thereby influencing the measurement result. Accordingly the pressure in the measurement branch should preferably be kept high, at least as high as the pressure in the process line at the location on which the sample is extracted, and during measurements the pressure shall be kept constant in the cuvette. Preferably, the pressure in the measuring branch exceeds the pressure in the process plant to ensure that dissolved air stays dissolved in the liquid food.

The method is specifically fitted for liquid food products such as raw milk or processed milk and other dairy products.

In one embodiment the measuring branch forms a closed system together with the process line. This means that the measuring branch only receives liquids flowing in the process conduit to which the measuring branch is connected.

Preferably, a regular (e.g. daily) cleaning of the measuring branch including the cuvette is performed when the dairy plant is subjected to the regular cleaning process and/or by flushing the branch with the same cleaning solutions used for cleaning the dairy plant. In an advantageous embodiment of the present invention at least one of the cleaning or rinsing liquids of the dairy may be used in the measuring branch for an adjustment, such as a standardization based on characteristics in the measured spectrum or spectra of the cleaning or rinsing liquids, especially characteristics originating from the appearance of ions belonging to the group comprising NO_3-ions and PO_4^{3-}. (By "standardization" is meant an adjustment of the instrument, (e.g. performed in the instrument software) made in order to make a plurality of spectrum measuring instruments performing in the same way so that copies of the same calibration software can be used on all the instruments and whereby all instruments will provide the same result when measuring the same sample.)

Preferably, the spectrometry is performed in the IR spectral range, e.g. in the MID-IR and/or the NIR spectral range, these ranges being specifically favourable for the analysis of milk.

Preferably, a spacer is arranged between the diamond windows and along the periphery of the measurement chamber, in order to define the correct distance (path length) between the windows as well as to support and stabilize the windows, when the chamber is flushed and filled with the liquid food. Preferably the opening in the spacer defining the light penetrating part of the windows has a diameter of less than 2.5 mm and preferably about 2 mm. It is preferred to have small windows because this will reduce the deflection when the chamber is exposed to high pressures. In an advantageous embodiment the spacer has a wedge form providing a change in width/height of spacer (equal to light path in chamber) of about 3 - 10 µm, e.g. about 6 µm across the 2 mm opening. Such wedge form will reduce or eliminate the tendency to the occurrence of internal reflections of the light beam inside the chamber.

Utility of the Invention

The improved method and the measurement cell therefore are intended for use in liquid food processing plants and more specifically dairies. In the following description the term milk includes raw milk and food products derived from that, and may include other kinds of liquid food products.

Advantages obtained by the invention:

The measurement takes place at normal process line pressure or even higher. Therefore, dissolved gases will not be released and accordingly, no air bubbles are produced. The measuring system is chemical resistant to all substances which stainless steel is resistant to. (This feature is an advantage during a regular cleaning of the food product processing system) The diamond windows ensure that the mechanical dimensions of the optical system are not subject to changes due to mechanical wear. The measuring system can operate in a process environment for a long time without any need for attended operators to perform zero adjustment, re-calibration, separate cleaning or other kind of maintenance operation on the flow system and IR measurement equipment. The measurement is an on-line measurement, always available to maintain an optimal production. Regularly, during normal operation, the flow system is "cleaned" by the samples themselves. Preferably, in a preferred embodiment, the normal process line cleaning procedure includes the measuring branch, i.e. the above mentioned flow system. Further, in the preferred embodiments, no "foreign" substances are introduced, i.e. no special cleaning or calibration agents are needed besides the cleaning and rinsing agents generally used in dairies. Therefore, a possible option comprises the return of the sample to the process line after the measurement.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a schematic diagram of an example of a measurement system and flow system according to the present invention.

Figure 2 is a schematic diagram of an example of a modified flow system according to the present invention.

Figure 3 is a schematic diagram of a third example of a flow system according to the present invention.

A high pressure pump 40, (e.g. a LPA/MSC50-h-pump as used in a FOSS ELECTRIC MILKOSCAN 50 or a single stroke pump providing a whole sample volume - e.g. 1.5 ml in one single stroke) provides the high pressure (e.g. about 400-500 bar). Typically at least a pressure of 200 bars is needed for homogenizing. Further the pump yield 40 will ensure a high flow rate through the IR-cuvette during a flushing period, so that the cuvette is cleaned by means of the flow rate of the milk, making further cleaning unnecessary for a number of hours. During the flushing period the pressure across the cuvette may reach 100 - 200 bars. To avoid degassing in the measurement period, the pressure of the measuring branch is maintained at at least the same pressure as the pressure at the location on which the sample is extracted from the process plant. Preferably the pressure in the measuring branch exceeds the pressure in the process plant. During a measurement the pressure is maintained at a substantially constant level by the use of a back pressure valve 88 as explained later.

In the embodiment shown in Fig. 1 an in-line filter 35 provides a filtered milk passing through the measurement branch comprising the cuvette 70. Optionally a valve 45 (Fig. 1) allows the milk to bypass the filter, the milk running directly towards waste 90. The high flow rate of milk along the inside of the filter 35 will provide a cleaning of the filter 35 when the valve 45 is open. To this end the valve 45 can be controlled by the control means 300. Preferably, the valve 45 also act as a safety valve which is set to open if the pressure exceeds e.g. 400 bars.

A homogenizer 50 (e.g. a S4000 as used in FOSS ELECTRIC MILKOSCAN 4000). A thorough homogenization of the liquid food product is needed in order to obtain a representative sample (a sample containing all components in the liquid food product) inside the very thin cuvette (typically having a width of 37-50 μm). A further reason for including homogenization is that the scattering of the infra red light passing through the cuvette depends on the particle size of the liquid sample. Accordingly a uniform homogenization is essential in order to have reproducible measurement conditions. The pressure drop across the homogenizer is about 200 bars. In the embodiments shown in Fig. 2 and 3 a safety valve also functioning as a contra valve 51 follows immediately after the homogenizer.

A further preheater or cooler 60, e.g. a coiled tube preferably wound on the periphery of a temperature stabilised IR cuvette, having an electrical resistor soldered to a copper body thermostatizing the milk sample to a predetermined temperature, e.g. to about 40 °C and preferably to 50 °C before entering the cuvette, and preferably comprising assigned temperature sensoring means connected to the control means 300 for controlling the temperature of the preheater or cooler. These controls and assignments are illustrated by phantom lines in Figure 1.

An IR cuvette 70, comprising a milk flow path and an IR light path crossing the milk flow path. The IR cuvette is part of an IR spectro-photo-meter allowing the analysis and/or quantitative determination of specific components of the milk in the IR cuvette. Preferably the IR cuvette structure includes a bypass milk flow path 82 (F... 2) for the auxiliary milk which is unable to pass through the very thin

carries the homogenised milk to a further preheater 60, the cuvette 70 and the back pressure valve 88 in the same way as in the other embodiments. In a modification of the embodiments shown in Fig. 1 - 3 the one way valve 23 is deleted as the one way function is inherently incorporated in the preferred homogenizer 50.

The optical system

The optical system 200 for measuring the IR absorption, preferably the MID-IR absorption, can be chosen between several known MID-IR spectrometric systems, and realised in several ways. Preferably a scanning interferometer, i.e. a FT-IR instrument is used, e.g. an IR-unit as used in FOSS ELECTRIC MILKOSCAN 120 . However, the optical system may instead include a filter wheel, comprising a plurality of IR filters appropriate for the desired measurements, e.g. as used in a FOSS ELECTRIC MILKOSCAN 50 or MILKOSCAN 102-104 and as described in GB-B-2 028 498, EP 0 012 492 and EP 0 629 290.

A simplified diagram of a suitable optical arrangement appears from Fig. 1 and Fig. 4. The Box 120 is an IR-source and scanning interferometer, 140 is a detector, and 160 is a computer. Scanning interferometers can be realised in several ways, cf., e.g. "Fourier Transform Infrared Spectrometry", Peter R. Griffiths and James A. de Haseth, John Wiley & Sons, 1986, and shall not be the topic of this application. The calculations for the determination of the quantities of the components in the milk are performed in the computer 160, and they are also well known to people in the art, e.g. as described in the above reference.

The IR cuvette

In the following the IR cuvette is described in detail. The IR-cuvette is designed for an optical path length of e.g. 37- 50 μm. The IR-cuvette must be made of strong materials, which are resistant to wear and pressure and resistant to mechanical and chemical influences, e.g. materials as used for the process plant itself. Preferably the IR cuvette is made from a stainless steel and diamond. Accordingly, the IR cuvette can be cleaned by the same liquid means which are used for the milk processing plant anyway in the dairy. Milk processing plants in dairies are regularly, e.g. daily/every 24 hours, flushed with several cleaning liquids, e.g. strong bases and/or acids.

The diamond and steel materials ensure that the cuvette will not be subjected to any noticeable wear. The physical dimensions and properties of the IR light path should be kept constant, preferably for the whole lifetime of the cuvette. Only the milk sample to be tested is changing. This is extremely important to the accuracy and reproducibility of the IR measurements.

An enlarged view of a first example of an embodiment of a cuvette is shown is Figure 5A and Figure 5B. An alternative embodiment of the IR cuvette is shown as a second example in Figures 6A, 6B, and 7. Figure 8 is a schematic top view which applies to both examples. Therefore the sections applied in Figures 5A, 5B, are shown on the schematic Figure 8 as well as the section C-C applied in

shown). The item 82 is an O-ring ensuring that no milk can intrude in the interface between the two cuvette parts.

In the preferred embodiment of the cuvette structure the bore holes 74 and openings 75, 77 are stepped, having stepwise or gradually decreasing diameters, in order to accomodate the inlet and outlet of liquid and IR light to the small measurement chamber 80 in the center of the cuvette.

Even in the enlarged scale view of Figures 5A, 5B, 6A, 6B and 7 the measurement chamber 80 containing the milk sample to be measured is hardly visible. The measurement chamber can be, e.g. about 2 - 3 mm in diameter and about 30 - 50 µm in width (equal to the optical path length). The actual dimensions in a cuvette in an apparatus according to the invention may depend on the milk or food product to be measured and the parameters wanted.

Figure 9 shows a section through the centre of the cuvette further enlarged in a large scale view, in order to show the inner details of a presently preferred embodiment. Milk flowing through the measuring branch enters the cuvette 70 through the tiny tube 78 at the bore 74 (Fig.5A) passing the adjacent bore 84 (Fig. 5A and Fig. 9), flows through the slot 89 into the measurement chamber 80 and exits from the cuvette through the opposite slot 89 and the bores 84, 74. Inside the cuvette this flow is forced into a thin (e.g. 30-50 µm thick) and relatively wide (e.g. 0.5 - 2 mm) flow passing the thin circular measurement chamber 80 between the diamond windows 73, 76. An optional bypass flow path 83 may be provided along the periphery of the windows 73, 76. If the track 83 is provided, preferably it can be restricted and possibly obstructed in order to ensure the measurement chamber is flushed thoroughly with the liquid to be measured. Figure 10 shows the spacer 81 and the adjacent track 83 and inlet and outlet bores 84. However in the presently preferred embodiment the cuvette has no bypass track 83.

However, the use of diamond windows may cause a serious problem. Due to the dielectic properties of diamond strong internal reflections of the IR-light beam may occur inside the cuvette. Such reflections will strongly influence the obtained spectrometric measurements and should preferably be avoided. Therefore, in an advantageous embodiment the spacer has a wedge form, i.e. having slightly inclined surfaces, providing a difference in light path of from 3 to 10 µm, preferably from 4 to 8 µm and most preferred about 6 µm, across the window opening for the IR light, thereby providing a light path varying e.g. from about 34 to 40 µm across the 2 mm opening. By this feature internal reflections may be eliminated or at least reduced. For sake of simplicity the wedge form is not illustrated in the drawings.

The control system

As it will be well known to people in the art such an optical system is delicate and has to be kept very

When the pump stops, a fraction of the new preheated sample is present in the cuvette. The pressure inside the cuvette will stay at a constant level, of about 10 - 20 bars preferably 12 bar, due to the back pressure valve 88. Preferably, the following step is about 25 sec's long. In this step the sample is analysed by the IR-spectrometer. In a further step a new sample is extracted from the fast loop. Preferably, the new sample is heated during the following about 25 sec's long step while a portion of the already heated (preceding) sample is measured in the cuvette. And so on. Accordingly, a new sample can be introduced approximately every 30 seconds, corresponding to about 120 measurements per hour.

By the embodiment shown in Fig.3 the pump 40 is a single stroke pump surrounded by heating means, e.g. a coil. The method is carried out as follows: In a first step of about two sec's a sample is sucked into the pump cylinder by one suction stroke of the piston. Then in a second step the sample is heated inside the pump cylinder for about 25 secs. In a third preferably short step of about 0.5 - 1.0 sec. duration the piston stroke is reversed, pushing the new sample through the homogenizer and partly through the cuvette and the back pressure valve. Thereby the old sample is flushed away either into a waste outlet 90 or returned into the fast loop and the process conduit. If the third step is performed in 0.5 sec. the flushing rate through the cuvette will be about 10 - 30 m/s with a displaced milk volume of about 1.5 ml. This flushing rate will ensure a thorough cleaning of the cuvette windows between each measurement. The advantage of the third embodiment is that the suction stroke may be slow (to avoid cavitation) and the reverse pumping stroke can be fast, thereby providing a high flushing rate and a better cleaning of the cuvette. Also in this embodiment a measurement rate of about 120 samples per hour is possible.

Due to the very stable and wear resistant properties of the cuvette calibrations and zero adjustments will normally only be necessary in the very first upstart of the equipment. However a routine check of the performance of the equipment may be carried out as follows. The measurement apparatus can be checked either

1) by application of a known calibration sample. The known sample is entered in the measurement branch by separating the flexible hose from the sample inlet at the fast loop and by dipping the flexible hose into a cup (110 in Fig. 2-3) filled with the known sample. A measuring cycle is executed and the result is noted and compared to the known sample data; and/or

2) by taking out a sample from the process stream at a point close to the measuring branch, e.g. at the fast-loop, possibly by separating the flexible hose from the pump 40, and collecting a sample in a cup, (110 in Fig. 2-3), bringing the sample to a reference instrument, e.g. a MilkoScan 50, 120 or system 4000 from Foss Electric A/S, and measuring the sample on the reference instrument.

3) by taking out a number (at least one) samples from the flow of already measured samples leaving the waste outlet 90 in Figure 1, bringing the sample to a reference instrument, e.g. a MilkoScan 50, 120 or system 4000 from Foss Electric A/S, and measuring the sample on the reference instrument. In order be able to compare the measured waste milk results to the test results from the cuvette the

cleaning procedures of the plant. This may be a so-called "CIP" (Clean In Place") procedure that uses heated acid and base solutions. During cleaning the typical flow rate will be below 10 m/s in the conduits of the milk plant but much higher in the cuvette. In an specifically advantageous embodiment of the present invention a number of the cleaning or rinsing liquids of the dairy are used as a calibration liquid for an adjustment or calibration of the optical part and specifically for an adjustment of the calculation and calibration software used for the calculation of the amounts of the tested components. A spectrum measured on a liquid containing, e.g. NO_3-ions and/or PO_4^{3-}-ions, may be used for an adjustment or calibration of the frequency/wavelength - axe.

As it appears from the foregoing pages a new reliable measurement method, flow system and cuvette has been provided, allowing almost continuous measurements on-line and in-line in diaries with the capacity of about 120 measurements pr. hour. It is obvious to people in the art that the different embodiments of the invention as described in the specification and shown in the drawings may be modified in several ways within the scope of the invention and accordingly the scope of protection is defined by the patent claims.

7. A flow system for in-line extraction of a sample stream from a liquid food processing plant, such as a dairy processing milk and milk products, and for carrying out the method according to any of the claims 1-6,
characterised in the flow system being directly connected to the liquid food processing plant, the flow system including a measurement cuvette (70) for spectrometric measurements for analysis of a liquid food product in the liquid food processing plant,
the flow system further comprising pump means (40) and back pressure valve means (88) to maintain a predetermined pressure inside the cuvette, said pressure being at least as high as the pressure inside the process line, and the cuvette having windows of a pressure resistant material.

8. A flow system according to claim 7, characterised in that the measurement cuvette has diamond windows (73, 76).

9. A flow system according to claim 7 or 8, characterised in that the pump means is able to induce a liquid by a flow rate of at least 10 and preferably 20 - 30 m/s into the cuvette (70) for a short period of at least 0.5 sec, said cuvette having a through flow area which is less than 0,5 mm^2, and preferably less than 0,10 mm^2. and most preferably less than 0,05 mm^2.

10. A flow system according to claim 7, 8 or 9, characterised in that the pump means is a single stroke pump (40 in Figure 1 and 3).

11. A flow system according to claim 7, characterised in that the measuring branch of the flowsystem (100) comprises a homogenizer (50) located upstream the cuvette (70), and at least one preheater (30, 60) located upstream the cuvette (70) and/or the homogenizer (50).

12. A flow system according to claim 7, characterised in that the measuring branch of the flowsystem (100) comprises a fast loop (12) for in-line extraction of the liquid food sample from the process tube (10) of the liquid food process plant, and that the measuring branch of the flow system (100) comprises an in-line filter (15) on the fast loop (12).

13. A flow system according to claim 7, characterised in that the preheater is a through flow heater/cooler (30) and that the measuring branch of the flow system (100) comprises an in-line filter (35) located downstream the preheater (30) and the pump (40) and upstream the homogenizer and the cuvette.

14. A flow system according to claim 7, characterised in that the measuring branch of the flow system (100) comprises a safety valve (45) located upstream the homogenizer (50) and the cuvette (70) said safety valve further being able to open under the control of control means (300).

23. Cuvette according to claims 21 or 22, c h a r a c t e r i s e d i n that an O-ring (82) is arranged along the periphery of the spacer, outside the spacer and/or along the outer periphery of the by pass flow path (83).

24. Cuvette according to claim 21, c h a r a c t e r i s e d i n that the spacer (81) is made of steel.

25. Cuvette according to claims 21, 23 or 24, c h a r a c t e r i s e d i n that the spacer (81) has wedge form, i.e. having slightly inclined surfaces, providing a difference in light path of from 3 to 10 μm, preferably from 4 to 8 μm and most preferred about 6 μm, across the window opening for the IR light.

26. Cuvette according to any of the claim 21-25, c h a r a c t e r i s e d i n that the spacer (81) is provided with slots (89) for the flow of liquid to and from the measurement chamber between the diamond windows in the centre of the cuvette.

27. Cuvette according to any of the claims 18-26, c h a r a c t e r i s e d i n that at least one of the boreholes (74, 75, 77) is stepped, i.e. having stepwise decreasing diameter from the outside towards the centre of the cuvette.

Fig. 2

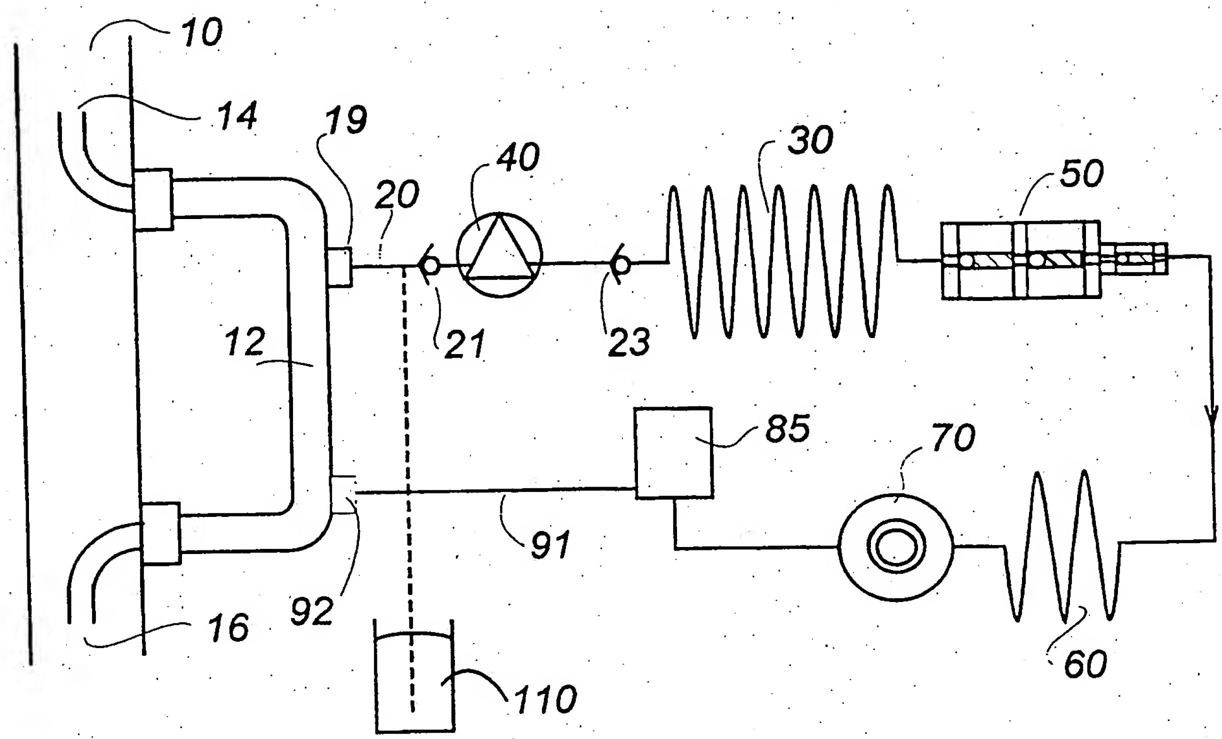

10
14
19
20
40
30
50
21
23
12
85
70
91
92
16
110
60

Fig. 3

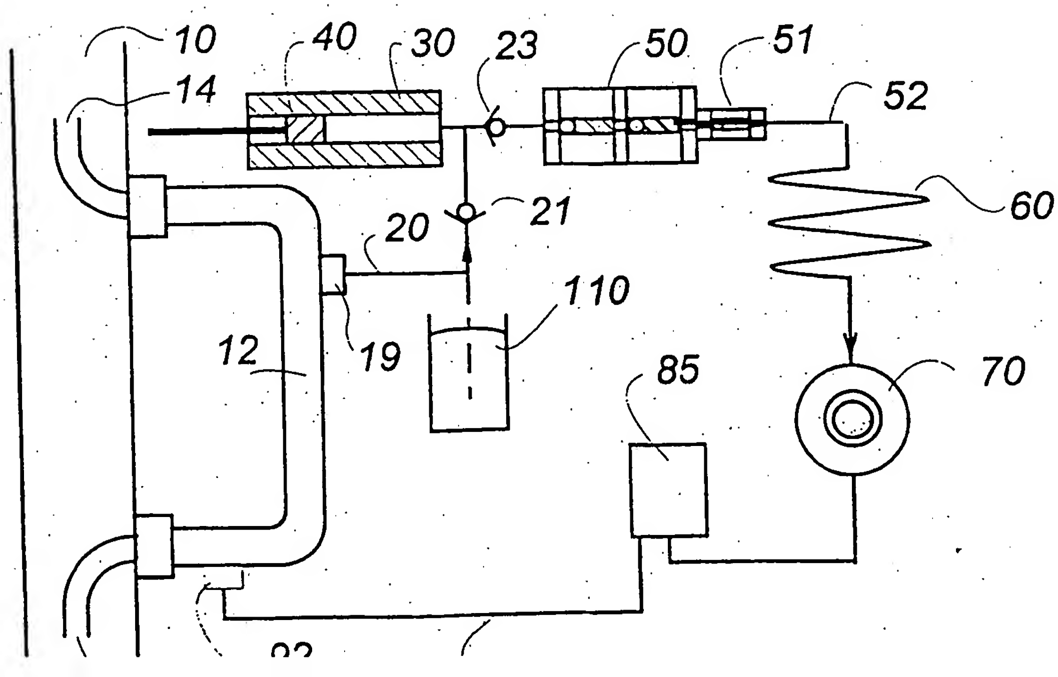

10
40
30
23
50
51
14
52
21
60
20
110
19
12
85
70

Fig. 6a

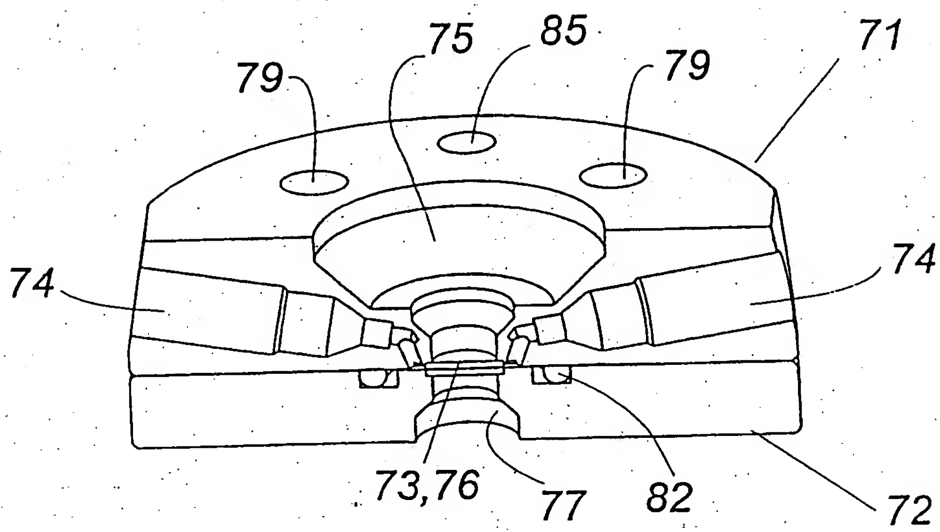

79
75
85
79
71
74
74
73,76
77
82
72

Fig. 6b

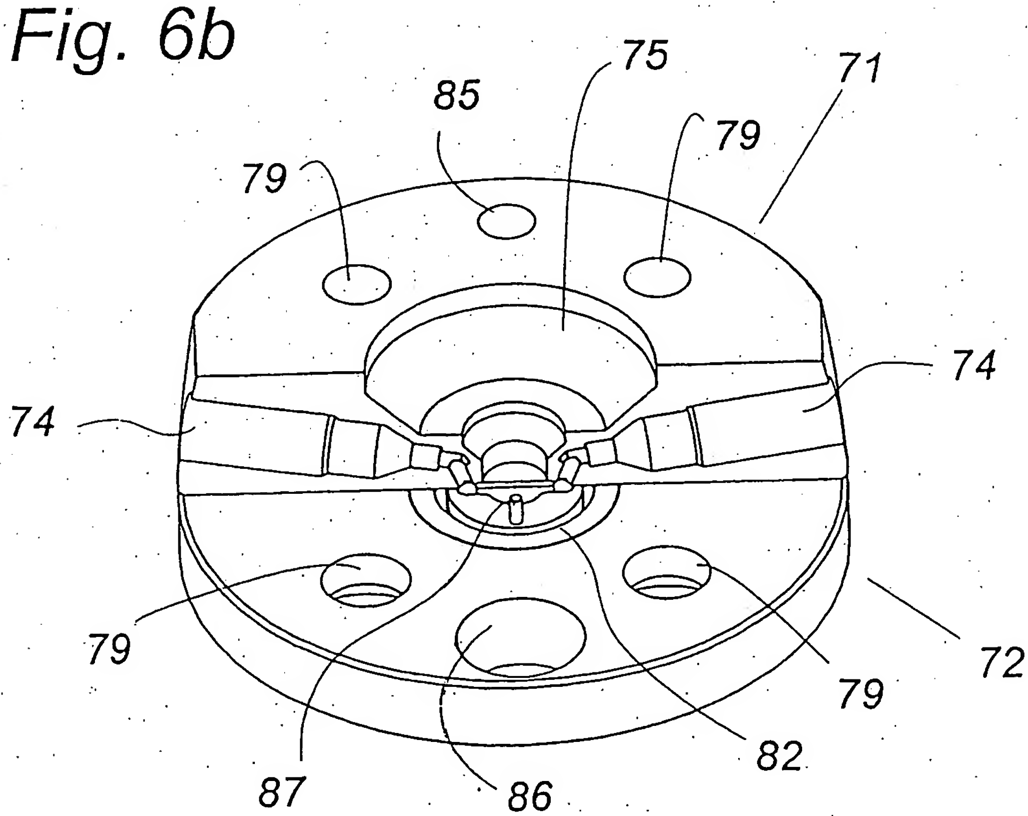

75
71
85
79
79
74
74
79
72
79
82
87
86

Fig. 9

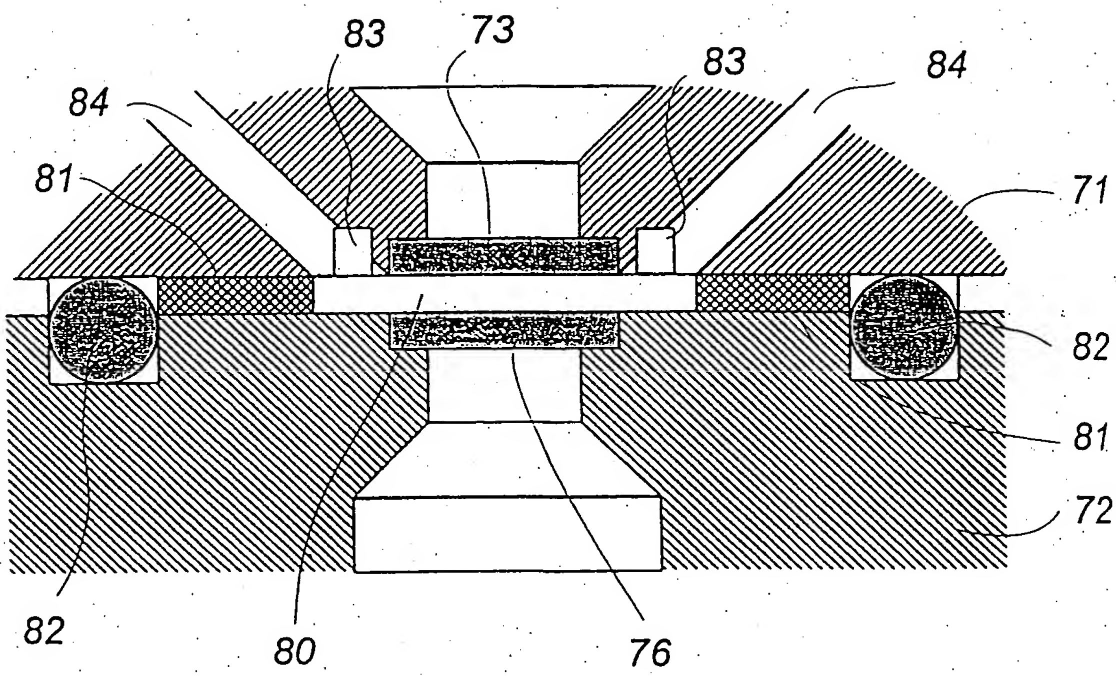
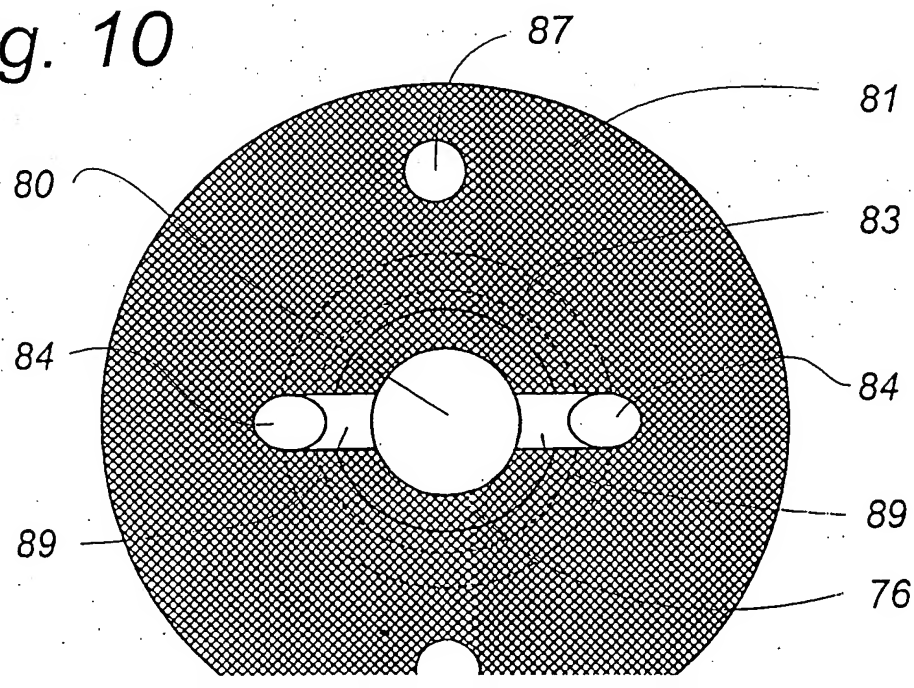


Fig. 10



INTERNATIONAL SEARCH REPORT

International application No.
PCT/DK 97/00492

A. CLASSIFICATION OF SUBJECT MATTER

IPC6: G01N 33/04, G01N 33/02, G01N 21/09 // G01N 21/35

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI, US PATENTS FULLTEXT, CA, FSTA, ANALYTICAL ABSTRACTS

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 4239394 A (POUL E. AEGIDIUS ET AL), 16 December 1980 (16.12.80), column 1, lines 24-36; column 4, lines 21-30; and the claims --	1-17
Y	Food Control, July 1990, Christopher Scotter, "Use of near infrared spectroscopy in the food industry with particular reference to its applications to on/in-line food processes", pages 142-149, page 146, right column, line 24 - page 148 --	1-17
Y	PACE/Process and control engineering, Volume 39, No 9, 1986, Richard Streamer, "Focus Feature, On-line near IR analysis", page 36, 38, 40, 42, 44; page 30, middle column, lines 4-7; Figure 1 --	1-17

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" erlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
18 February 1998	24 -02- 1998

INTERNATIONAL SEARCH REPORT

Information on patent family members

03/02/98

International application No.
PCT/DK 97/00492

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4239394 A	16/12/80	JP 1123942 C	30/11/82
		JP 54009688 A	24/01/79
		JP 57012091 B	09/03/82
GB 1305214 A	31/01/73	CH 515505 A	15/11/71
		DE 2053380 A	22/06/72
		NL 7016172 A	08/05/72
US 5003174 A	26/03/91	DE 3822445 A,C	04/01/90
US 4910403 A	20/03/90	CA 2001615 A	02/05/90
US 5046854 A	10/09/91	NONE	